Double Conjugate Addition of Dithiols to Propargylic Carbonyl Systems To Generate Protected 1,3-Dicarbonyl Compounds

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 'C19 H29 N O3 S3'
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 '-x+y, -x, z-1/3'

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multi-scan from symmetry-related measurements
 Sortav (Blessing 1995)
;

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;
 The absolute structure was determined (Flack parameter 0.05(7)).
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 HKL Denzo and Scalepack (Otwinowski & Minor 1997)
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_computing_structure_solution     'SIR-92 (Altomare et al. 1994)'
_computing_structure_refinement   'SHELXL-97 (Sheldrick 1997)'
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 Refinement of F^2^ against ALL reflections.  The weighted R-factor wR and
 goodness of fit S are based on F^2^, conventional R-factors R are based
 on F, with F set to zero for negative F^2^. The threshold expression of
 F^2^ > 2sigma(F^2^) is used only for calculating R-factors(gt) etc. and is
 not relevant to the choice of reflections for refinement.  R-factors based
 on F^2^ are statistically about twice as large as those based on F, and R-
 factors based on ALL data will be even larger.
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S2 S 0.08376(8) 0.31319(10) 0.02038(6) 0.0498(2) Uani 1 1 d . . .
S3 S 0.64462(7) 0.41427(7) -0.04545(4) 0.03173(16) Uani 1 1 d . . .
O1 O 0.2498(2) 0.1518(2) -0.01520(14) 0.0421(5) Uani 1 1 d . . .
O2 O 0.6517(2) 0.2870(2) -0.02197(14) 0.0413(5) Uani 1 1 d . . .
O3 O 0.6917(2) 0.4743(3) -0.12513(12) 0.0413(5) Uani 1 1 d . . .
N1 N 0.4751(2) 0.3743(2) -0.03469(14) 0.0306(5) Uani 1 1 d . . .
C1 C 0.3774(3) 0.2603(3) 0.02362(18) 0.0342(6) Uani 1 1 d . . .
H1 H 0.4308 0.2136 0.0481 0.041 Uiso 1 1 calc R . .
C2 C 0.3268(3) 0.3229(3) 0.09094(18) 0.0347(6) Uani 1 1 d . . .
H2B H 0.4101 0.3802 0.1280 0.042 Uiso 1 1 calc R . .
H2C H 0.2496 0.2408 0.1225 0.042 Uiso 1 1 calc R . .
C3 C 0.2675(3) 0.4204(3) 0.06205(17) 0.0331(6) Uani 1 1 d . . .
C4 C 0.3793(3) 0.5369(3) 0.00432(17) 0.0337(6) Uani 1 1 d . . .
H4A H 0.3358 0.5933 -0.0185 0.040 Uiso 1 1 calc R . .
H4B H 0.4661 0.6056 0.0364 0.040 Uiso 1 1 calc R . .
C5 C 0.4307(3) 0.4792(3) -0.06608(17) 0.0309(5) Uani 1 1 d . . .
H5 H 0.5213 0.5649 -0.0882 0.037 Uiso 1 1 calc R . .
C6 C 0.2761(4) 0.0706(4) -0.0758(2) 0.0527(9) Uani 1 1 d . . .
H6A H 0.1828 -0.0130 -0.0931 0.079 Uiso 1 1 calc R . .
H6B H 0.3389 0.0355 -0.0533 0.079 Uiso 1 1 calc R . .
H6C H 0.3247 0.1338 -0.1226 0.079 Uiso 1 1 calc R . .
C7 C 0.0391(5) 0.4550(6) 0.0361(3) 0.0696(12) Uani 1 1 d . . .
H7A H 0.0892 0.5327 -0.0051 0.084 Uiso 1 1 calc R . .
H7B H -0.0677 0.4140 0.0299 0.084 Uiso 1 1 calc R . .
C8 C 0.0855(5) 0.5190(5) 0.1195(3) 0.0638(10) Uani 1 1 d . . .
H8A H 0.0039 0.4649 0.1581 0.077 Uiso 1 1 calc R . .
H8B H 0.1093 0.6224 0.1190 0.077 Uiso 1 1 calc R . .
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C9 C 0.3238(4) 0.4141(4) -0.13708(17) 0.0387(6) Uani 1 1 d . . .
H9A H 0.2456 0.3147 -0.1217 0.046 Uiso 1 1 calc R . .
H9B H 0.2773 0.4744 -0.1461 0.046 Uiso 1 1 calc R . .
C10 C 0.3918(4) 0.4039(4) -0.21671(19) 0.0488(8) Uani 1 1 d . . .
H10 H 0.4522 0.3572 -0.2053 0.059 Uiso 1 1 calc R . .
C11 C 0.2720(5) 0.3070(5) -0.2768(2) 0.0662(12) Uani 1 1 d . . .
H11A H 0.3163 0.3075 -0.3291 0.099 Uiso 1 1 calc R . .
H11B H 0.2046 0.3447 -0.2840 0.099 Uiso 1 1 calc R . .
H11C H 0.2181 0.2068 -0.2558 0.099 Uiso 1 1 calc R . .
C12 C 0.4902(5) 0.5527(5) -0.2527(2) 0.0628(11) Uani 1 1 d . . .
H12A H 0.5262 0.5419 -0.3055 0.094 Uiso 1 1 calc R . .
H12B H 0.5732 0.6091 -0.2163 0.094 Uiso 1 1 calc R . .
H12C H 0.4351 0.6041 -0.2597 0.094 Uiso 1 1 calc R . .
C13 C 0.7515(3) 0.5566(3) 0.02371(15) 0.0297(5) Uani 1 1 d . . .
C14 C 0.7385(3) 0.5275(3) 0.10620(16) 0.0324(6) Uani 1 1 d . . .
H14 H 0.6728 0.4320 0.1258 0.039 Uiso 1 1 calc R . .
C15 C 0.8224(3) 0.6394(3) 0.15964(16) 0.0363(6) Uani 1 1 d . . .
H15 H 0.8132 0.6199 0.2163 0.044 Uiso 1 1 calc R . .
C16 C 0.9202(3) 0.7800(3) 0.13252(17) 0.0373(6) Uani 1 1 d . . .
C17 C 0.9327(4) 0.8048(3) 0.04942(18) 0.0426(7) Uani 1 1 d . . .
H17 H 1.0004 0.8996 0.0297 0.051 Uiso 1 1 calc R . .
C18 C 0.8495(4) 0.6958(3) -0.00499(16) 0.0393(7) Uani 1 1 d . . .
H18 H 0.8589 0.7154 -0.0616 0.047 Uiso 1 1 calc R . .
C19 C 1.0135(5) 0.9002(4) 0.1910(2) 0.0558(9) Uani 1 1 d . . .
H19A H 0.9517 0.9052 0.2339 0.084 Uiso 1 1 calc R . .
H19B H 1.0634 0.9938 0.1621 0.084 Uiso 1 1 calc R . .
H19C H 1.0864 0.8801 0.2154 0.084 Uiso 1 1 calc R . .

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S3 0.0290(3) 0.0373(4) 0.0347(3) -0.0052(3) -0.0001(3) 0.0210(3)
O1 0.0349(11) 0.0272(10) 0.0597(13) -0.0079(9) -0.0021(9) 0.0122(9)
O2 0.0398(12) 0.0389(12) 0.0567(12) -0.0073(10) -0.0024(9) 0.0283(10)
O3 0.0375(11) 0.0545(13) 0.0334(9) -0.0056(9) 0.0041(8) 0.0242(11)
N1 0.0271(11) 0.0314(12) 0.0384(11) 0.0009(9) 0.0000(9) 0.0185(10)
C1 0.0327(15) 0.0265(13) 0.0439(14) -0.0001(11) -0.0008(12) 0.0153(12)
C2 0.0298(14) 0.0308(14) 0.0424(13) 0.0042(11) 0.0052(11) 0.0142(12)
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C4 0.0336(15) 0.0280(14) 0.0421(14) -0.0025(11) 0.0005(11) 0.0173(12)
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C6 0.057(2) 0.0410(19) 0.067(2) -0.0191(16) -0.0157(17) 0.0291(17)
C7 0.058(2) 0.085(3) 0.091(3) -0.001(2) 0.000(2) 0.055(3)
C8 0.059(2) 0.062(2) 0.088(3) 0.003(2) 0.017(2) 0.044(2)
C9 0.0403(16) 0.0446(17) 0.0415(14) -0.0037(13) -0.0072(12) 0.0290(15)
C10 0.058(2) 0.069(2) 0.0396(15) -0.0097(15) -0.0088(14) 0.047(2)
C11 0.082(3) 0.093(3) 0.0515(19) -0.030(2) -0.0283(19) 0.065(3)
C12 0.060(2) 0.098(3) 0.0470(18) 0.010(2) 0.0113(16) 0.052(2)
C13 0.0270(13) 0.0331(14) 0.0330(12) 0.0013(10) 0.0010(10) 0.0181(11)
C14 0.0287(13) 0.0337(15) 0.0328(12) 0.0058(11) 0.0034(10) 0.0141(12)
C15 0.0352(15) 0.0397(16) 0.0307(12) 0.0039(11) 0.0013(11) 0.0163(13)
C16 0.0377(16) 0.0341(15) 0.0379(14) -0.0013(11) -0.0026(12) 0.0162(13)
C17 0.0446(18) 0.0318(15) 0.0422(15) 0.0077(12) 0.0033(13) 0.0123(14)
C18 0.0466(17) 0.0388(16) 0.0302(12) 0.0071(11) 0.0048(12) 0.0196(14)
C19 0.060(2) 0.0432(19) 0.0474(16) -0.0056(14) -0.0060(15) 0.0130(17)

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 All esds (except the esd in the dihedral angle between two l.s. planes)
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 are estimated using the full covariance matrix.  The cell esds are taken
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S3 O3 1.434(2) . ?
S3 O2 1.436(2) . ?
S3 N1 1.630(2) . ?
S3 C13 1.770(3) . ?
O1 C1 1.412(4) . ?
O1 C6 1.431(4) . ?
N1 C1 1.481(4) . ?
N1 C5 1.494(3) . ?
C1 C2 1.519(4) . ?
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C2 C3 1.524(4) . ?
C2 H2B 0.9900 . ?
C2 H2C 0.9900 . ?
C3 C4 1.536(4) . ?
C4 C5 1.530(4) . ?
C4 H4A 0.9900 . ?
C4 H4B 0.9900 . ?
C5 C9 1.529(4) . ?
C5 H5 1.0000 . ?
C6 H6A 0.9800 . ?
C6 H6B 0.9800 . ?
C6 H6C 0.9800 . ?
C7 C8 1.501(7) . ?
C7 H7A 0.9900 . ?
C7 H7B 0.9900 . ?
C8 H8A 0.9900 . ?
C8 H8B 0.9900 . ?
C9 C10 1.525(4) . ?
C9 H9A 0.9900 . ?
C9 H9B 0.9900 . ?
C10 C12 1.506(6) . ?
C10 C11 1.527(5) . ?
C10 H10 1.0000 . ?
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C12 H12B 0.9800 . ?
C12 H12C 0.9800 . ?
C13 C14 1.385(4) . ?
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C14 C15 1.382(4) . ?
C14 H14 0.9500 . ?
C15 C16 1.391(4) . ?
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C16 C19 1.504(4) . ?
C17 C18 1.374(4) . ?
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O3 S3 N1 107.89(12) . . ?
O2 S3 N1 107.00(12) . . ?
O3 S3 C13 106.34(13) . . ?
O2 S3 C13 108.15(13) . . ?
N1 S3 C13 107.53(12) . . ?
C1 O1 C6 114.2(2) . . ?
C1 N1 C5 118.6(2) . . ?
C1 N1 S3 120.59(18) . . ?
C5 N1 S3 118.73(18) . . ?
O1 C1 N1 111.1(2) . . ?
O1 C1 C2 106.4(2) . . ?
N1 C1 C2 111.9(2) . . ?
O1 C1 H1 109.1 . . ?
N1 C1 H1 109.1 . . ?
C2 C1 H1 109.1 . . ?
C1 C2 C3 114.7(2) . . ?
C1 C2 H2B 108.6 . . ?
C3 C2 H2B 108.6 . . ?
C1 C2 H2C 108.6 . . ?
C3 C2 H2C 108.6 . . ?
H2B C2 H2C 107.6 . . ?
C2 C3 C4 108.9(2) . . ?
C2 C3 S2 111.6(2) . . ?
C4 C3 S2 114.7(2) . . ?
C2 C3 S1 107.3(2) . . ?
C4 C3 S1 109.89(19) . . ?
S2 C3 S1 104.19(14) . . ?
C5 C4 C3 115.7(2) . . ?
C5 C4 H4A 108.3 . . ?
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C5 C4 H4B 108.3 . . ?
C3 C4 H4B 108.3 . . ?
H4A C4 H4B 107.4 . . ?
N1 C5 C9 111.5(2) . . ?
N1 C5 C4 109.7(2) . . ?
C9 C5 C4 115.5(2) . . ?
N1 C5 H5 106.5 . . ?
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H6B C6 H6C 109.5 . . ?
C8 C7 S2 110.2(3) . . ?
C8 C7 H7A 109.6 . . ?
S2 C7 H7A 109.6 . . ?
C8 C7 H7B 109.6 . . ?
S2 C7 H7B 109.6 . . ?
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C7 C8 H8A 109.5 . . ?
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C7 C8 H8B 109.5 . . ?
S1 C8 H8B 109.5 . . ?
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C9 C10 H10 108.1 . . ?
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C10 C11 H11B 109.5 . . ?
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H11A C11 H11C 109.5 . . ?
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H12A C12 H12C 109.5 . . ?
H12B C12 H12C 109.5 . . ?
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C17 C18 H18 120.4 . . ?
C13 C18 H18 120.4 . . ?
C16 C19 H19A 109.5 . . ?
C16 C19 H19B 109.5 . . ?
H19A C19 H19B 109.5 . . ?
C16 C19 H19C 109.5 . . ?
H19A C19 H19C 109.5 . . ?
H19B C19 H19C 109.5 . . ?

loop_
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 _geom_torsion_atom_site_label_2
 _geom_torsion_atom_site_label_3
 _geom_torsion_atom_site_label_4
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 _geom_torsion_site_symmetry_2
 _geom_torsion_site_symmetry_3
 _geom_torsion_site_symmetry_4
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 _geom_torsion_publ_flag
O3 S3 N1 C1 155.1(2) . . . . ?
O2 S3 N1 C1 25.4(2) . . . . ?
C13 S3 N1 C1 -90.6(2) . . . . ?
O3 S3 N1 C5 -41.6(2) . . . . ?
O2 S3 N1 C5 -171.3(2) . . . . ?
C13 S3 N1 C5 72.7(2) . . . . ?
C6 O1 C1 N1 63.6(3) . . . . ?
C6 O1 C1 C2 -174.3(3) . . . . ?
C5 N1 C1 O1 72.0(3) . . . . ?
S3 N1 C1 O1 -124.7(2) . . . . ?
C5 N1 C1 C2 -46.8(3) . . . . ?
S3 N1 C1 C2 116.5(2) . . . . ?
O1 C1 C2 C3 -72.6(3) . . . . ?
N1 C1 C2 C3 48.9(3) . . . . ?
C1 C2 C3 C4 -51.4(3) . . . . ?
C1 C2 C3 S2 76.1(3) . . . . ?
C1 C2 C3 S1 -170.3(2) . . . . ?
C7 S2 C3 C2 157.7(2) . . . . ?
C7 S2 C3 C4 -77.9(3) . . . . ?
C7 S2 C3 S1 42.2(2) . . . . ?
C8 S1 C3 C2 -147.0(2) . . . . ?
C8 S1 C3 C4 94.8(2) . . . . ?
C8 S1 C3 S2 -28.6(2) . . . . ?
C2 C3 C4 C5 52.4(3) . . . . ?
S2 C3 C4 C5 -73.3(3) . . . . ?
S1 C3 C4 C5 169.7(2) . . . . ?
C1 N1 C5 C9 -82.9(3) . . . . ?
S3 N1 C5 C9 113.5(2) . . . . ?
C1 N1 C5 C4 46.3(3) . . . . ?
S3 N1 C5 C4 -117.3(2) . . . . ?
C3 C4 C5 N1 -49.2(3) . . . . ?
C3 C4 C5 C9 77.8(3) . . . . ?
C3 S2 C7 C8 -45.6(3) . . . . ?
S2 C7 C8 S1 30.1(4) . . . . ?
C3 S1 C8 C7 0.0(3) . . . . ?
N1 C5 C9 C10 -73.3(3) . . . . ?
C4 C5 C9 C10 160.6(3) . . . . ?
C5 C9 C10 C12 -69.7(3) . . . . ?
C5 C9 C10 C11 167.1(3) . . . . ?
O3 S3 C13 C14 -174.9(2) . . . . ?
O2 S3 C13 C14 -45.5(2) . . . . ?
N1 S3 C13 C14 69.7(2) . . . . ?
O3 S3 C13 C18 4.0(3) . . . . ?
O2 S3 C13 C18 133.4(2) . . . . ?
N1 S3 C13 C18 -111.3(2) . . . . ?
C18 C13 C14 C15 1.1(4) . . . . ?
S3 C13 C14 C15 -180.0(2) . . . . ?
C13 C14 C15 C16 -0.4(4) . . . . ?
C14 C15 C16 C17 -0.8(5) . . . . ?
C14 C15 C16 C19 -178.6(3) . . . . ?
C15 C16 C17 C18 1.4(5) . . . . ?
C19 C16 C17 C18 179.2(3) . . . . ?
C16 C17 C18 C13 -0.8(5) . . . . ?
C14 C13 C18 C17 -0.5(4) . . . . ?
S3 C13 C18 C17 -179.4(3) . . . . ?

_diffrn_measured_fraction_theta_max    0.997
_diffrn_reflns_theta_full              27.48
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_refine_diff_density_max    0.423
_refine_diff_density_min   -0.224
_refine_diff_density_rms    0.053
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